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SUFFIELD MEMORANDUM

NO. 1104

AN INVESTIGATION OF THE SUITABILITY OF USING A SIEGLAFF-MCKELVEY HIGH SHEAR RATE MELT RHEOMETER FOR THE MEASUREMENT OF LOW VISCOSITY POLYMER SOLUTIONS (U)

by

J.B. Hacker\*

PCN No. 13E10

August 1984





\* Co-operative student from the University of British Columbia, May — August 1983

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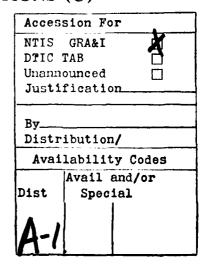
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#### **PREFACE**

The following report was prepared by Mr. J.B. Hacker as part of the requirement for co-operative student education in the Faculty of Engineering, University of British Columbia. The work was performed under the direction of Dr. S.J. Armour at the Defence Research Establishment Suffield during May — August, 1983.

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#### **ABSTRACT**

An investigation was made into the adaptation of a Sieglaff-McKelvey high shear rate melt rheometer for use in measuring low viscosity fluids. Using calibrated Newtonian oils, viscosities as low as 1.5 poise were found to be measurable with better than 5% accuracy over a shear rate range from a nominal 1000 1/S to 100,000 1/S at temperatures as low as 5%C above ambient. However, difficulties were experienced in the measurement of low viscosity polymer solutions. With the stock capillaries 25.40 mm in length, a combination of wall slip and entrance/exit effects made calculating true viscosities impossible. It was concluded that capillaries with a diameter of 1.0 mm or greater and variable lengths of up to 50 mm would be required to eliminate slip and still obtain a pressure drop within the measurable range of the instrument. To facilitate accurate measurement of low pressure drops, a new piston design was proposed using low-friction, low leakage Teflon o-rings. This would also expand the lower limits of the instrument's viscosity and shear rate ranges.

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#### **INTRODUCTION**

1. The Sieglaff-McKelvey Capillary Rheometer marketed by Imass Inc. is a commercial instrument intended for the measurement of the viscosity of polymer melts at high shear rates and at temperatures well above ambient. Because the interests of the Physical Chemistry Group at the Defence Research Establishment Suffield (DRES) lie in the behaviour of low-viscosity polymer fluids approximately two to three orders of magnitude thinner than melts and at temperatures much closer to ambient, an investigation was made into the suitability of using this rheometer to measure such fluids. It was hoped not only to determine the applicability of using this instrument for low-viscosity polymer solutions, but also to determine the limits of viscosity, shear rate, and temperature ranges of the instrument.

#### INSTRUMENT DESCRIPTION

- 2. The Sieglaff-McKelvey High Shear Rate Capillary Rheometer uses the principle of flow through a tube to measure fluid viscosities. It is a two piece instrument consisting of a rigid cast iron loading frame and a control and recording console (Figure 1).
- 3. The loading frame houses a pneumatically operated piston which fits into a replaceable stainless steel cylinder barrel. A number of piston diameters are available from 6.35 mm to 12.70 mm, the largest of which requires approximately 10 mL of fluid (Appendix A). Small diameter capillaries of 25.40 mm in length are screwed tightly into the bottom of the cylinder barrel. A selection of capillary diameters are available from 0.30 mm to 1.80 mm and are constructed out of pressed in precision bore tubing (Figure 2). The diameters are calibrated to 5 digit accuracy at the factory (Appendix B).
- 4. The control console is divided into three main sub-sections: (1) piston drive control; (2) temperature regulation; (3) the recording system.
- 5. The piston drive consists of a pneumatic valve to control the direction of piston travel and a pressure regulator and gauge to regulate the force exerted by the piston when in the constant stress mode. Alternatively, a needle valve on the loading frame may be used to control piston velocity in the constant rate mode.
- 6. The electronic temperature regulator contains potentiometers to pre-set the desired temperature and a digital display to indicate either pre-set or sample temperature. Temperature control is affected by changing the duty cycle of the resistance-type elements.
- 7. The recording system consists of a high speed Hewlett-Packard Model 7402A 2-pen oscillographic recorder for a permanent record of both piston velocity and piston load. Two piston load cells are available covering a range from 0-900 N and 0-9000 N, and are easily interchanged.
- 8. The two bench top units are interconnected by electrical cables and pressure hoses for the pneumatic drive. The instrument requires clean compressed air or nitrogen at 6 to 7 bar pressure for dependable operation.

#### **THEORY**

#### 1. General Concepts

- 9. In rheology, the study of the flow and deformation of materials, the most broad characterization of fluids is whether they are Newtonian or Non-Newtonian.
- 10. Newtonian fluids are typified by low molecular mass liquids such as water, oils and most solvents. The shear viscosity of a Newtonian fluid is dependent on temperature only; the higher the temperature, the thinner, or less viscous, the fluid. This change in viscosity with temperature follows approximately Andrade's law which for Newtonian fluids is (Reference 1):

$$\eta = ae^{-b/T}$$
 [1]

Because of this sensitivity, viscosities are always quoted at a specific temperature. The viscosity of Newtonian fluids in steady shear is easily measured on a number of very accurate, simple, commercial instruments. As a result these fluids are often used as calibrating standards for more complex rheometers.

Non-Newtonian fluids, however, are somewhat more complicated. These fluids exhibit shear dependent viscosity and most are either shear thickening or shear thinning. That is, their viscosity at a constant temperature either increases or decreases as the shear rate is increased. Thus, to characterize the viscosity flow curve for a Non-Newtonian fluid, it is necessary to measure the viscosity over a wide range of shear rates at the desired temperature. Shear rates are measured in units of reciprocal seconds (1/S), and the ability to measure the range from 0 to 100,000 1/S is generally required to fully characterize a fluid. At the upper and lower extremes of the shear rate range, the viscosity of most Non-Newtonian fluids approaches limiting viscosities known respectively as the infinite shear viscosity and the zero shear viscosity (Figure 3). Since most fluids of interest to DRES are Non-Newtonian in behaviour, it was hoped that the Sieglaff-McKelvey Capillary Rheometer could be used to determine infinite shear viscosities by measuring viscosities in the upper shear rate range from approximately 1000 1/S to 100,000 1/S, a range unobtainable with other instruments in the laboratory.

#### II. Capillary Flow Viscometry — Non-Newtonian Fluids

12. To determine shear viscosity, it is necessary to evalute both the shear stress and the shear rate at some convenient point in the capillary, usually the wall. The classical formula for shear stress at the wall of a capillary is the so-called "Poiseuille Law" (Reference 2):

$$\tau_{\omega} = \frac{R_c \Delta P}{2L_c}$$
 [2]

where  $\tau_{\alpha i}$  = shear stress at the wall

 $\Delta P$  = pressure drop due to viscous losses

 $R_c = capillary radius$ 

 $L_c$  = capillary length

- 13. To be valid this equation requires steady laminar flow with straight flow streamlines and no change in velocity in the direction of flow. Because such idealistic assumptions are rarely met in working capillary rheometers, particularly with Non-Newtonian fluids, several corrections are typically employed.
- 14. The most important correction which takes into account both the entrance and exit flow losses and the elastic potential energy acquired by the fluid during steady flow can be evaluated using a method first developed by Bagley (Reference 3). This method determines an equivalent extra length (L<sub>c</sub>) of tube which would have to be added to the actual length if the total measured pressure drop were that for an entirely fully developed region.
- 15. Since from equation [2]

$$\Delta P = 2 \tau_{\omega} \left( \frac{L_c}{R_c} + \frac{L_c}{R_c} \right)$$
 [3]

and noting shear stress  $(\tau_{\omega})$  is a unique function of shear rate, the end correction  $(L_c/R_c)$  can be evaluated for Non-Newtonian fluids by plotting pressure drop versus  $L_c/R_c$  using data from a number of different capillaries at a constant shear rate and extrapolating back to the x-axis to obtain the equivalent extra length  $(L_c)$ . It should be noted that this is usually done using a constant capillary radius  $(R_c)$  with variable

lengths (L<sub>c</sub>) to obtain different values of  $L_c/R_c$ . However due to the design of the Sieglaff-McKelvey Rheometer, the lengths were not variable and different radii had to be used instead.

- 16. A second correction must often be made for the kinetic energy gain in the fluid as it is accelerated from rest to its exit velocity at the end of the capillary. However, due to the low flow rates employed by the Sieglaff-McKelvey, error due to this effect was below 0.05% and considered negligible.
- 17. The classical wall shear rate equation for Newtonian fluids having a fully developed parabolic velocity profile within the capillary is given by (Reference 2):

$$\Gamma = \frac{4Q}{\pi R_{\perp}^3}$$
 [4]

where  $\Gamma$  = Newtonian wall shear rate

Q = flow rate

R<sub>c</sub> = capillary radius

and (Reference 4):

$$Q = \frac{V_p}{\pi R_p^2}$$
 [5]

where  $V_p = piston velocity$ 

 $R_p$  = piston radius

18. For Non-Newtonian fluids, the velocity profile will not be a true parabola and a correction known as the Rabinowitsch equation must be applied as follows (Reference 2):

$$\dot{\gamma}_{\omega} = \left[ \frac{3n' + 1}{4n'} \right] \Gamma \tag{6}$$

where  $\dot{y}_{(i)}$  = wall shear rate

and 
$$n' = \frac{d\ln \tau_{\omega}}{d\ln \Gamma}$$
 [7]

19. An additional problem with Non-Newtonian fluids is slip at the wall of the capillary. For true laminar flow within the capillary, the velocity profile must be a smooth curve with a maximum velocity at the centre and zero velocity at the wall. When slip occurs the velocity is no longer zero at the wall but some finite value (Figure 4). This effect may be accounted for by assuming an effective slip velocity superimposed upon the fluid in the tube and using a revised version of the Rabinowitsch equation employing a no-slip shear rate (Reference 2):

$$\Gamma_{no\ slip} = \Gamma_{measured} - \frac{4\beta\tau_{\omega}}{R_c}$$
 [8]

where  $\beta$ , the slip-coefficient, is the slope of the graph of  $\Gamma/4\tau_{\omega}$  versus  $1/R_c$  and a function of shear stress.

- 20. It should be noted that the slip correction may not be applied if the end correction is not negligible. As a general rule of thumb, slip can be avoided simply by using capillaries of sufficiently large diameter, typically greater than 1.0 mm.
- 21. From the values of shear stress and shear rate at the wall, the viscosity can be calculated using the equation (Reference 2):

$$\eta = \frac{\tau_{\omega}}{\dot{\gamma}_{\omega}}$$
 [9]

where  $\eta = \text{viscosity}$ 

22. Thus by calculating the viscosity over a range of shear rates, a plot of viscosity versus shear rate of the test fluid may be constructed and any limiting viscosity easily determined.

#### **EXPERIMENTAL**

23. As an initial attempt to gauge the accuracy of the rheometer, and to determine the lower limit of its viscosity range, a series of tests were run using Newtonian oils of known viscosity at  $40.0^{\circ}$ C. Viscosities ranged from 200 poise to 1.5 poise and capillary diameters from 0.30 to 0.60 mm were used. From the data recorded on the strip chart, plots of pressure drop ( $\Delta P$ ) versus shear rate ( $\Gamma$ ) were made and the best straight line fitted using the least squares method. A typical plot is shown in Figure 5. From the

equation of this line and the capillary dimensions viscosities were calculated and compared with the certified National Bureau of Standards (NBS) viscosity at the same temperature (Appendix C and Figure 6).

- 24. From these runs it was determined that viscosities as low as 1 poise were measurable, though less accurately, as the pressure drops became masked by inconsistencies such as piston friction and leakage. In all cases accuracy was better than 5%.
- 25. It was also determined that Newtonian shear rates (Γ) from a nominal 1000 1/S to 100,000 1/S were obtainable, the lower limit being an inverse function of the test sample viscosity and ranging from 200 1/S for the 200 poise oil to 2000 1/S for the 1.5 poise oil.
- 26. As viscosity for most fluids is very sensitive to temperature, extensive testing and calibration of the temperature regulation was done. A calibrated digital thermocouple (Cole-Parmer model 8502-25) accurate to  $\pm$  0.05°C was used to measure temperatures within the cylinder barrel. Results indicated a temperature variation of  $\pm$  0.3°C along the length of the cylinder barrel from the value as read on the digital display of the control console.
- 27. A thermocouple was also inserted into the capillary itself, and variations of less than  $\pm$  0.1°C were noted. Thus, temperature regulation, once calibrated, was judged to be adequate for the accuracy expected from the instrument ( $\pm$  5%).
- 28. With the basic limits of the rheometer now established, a representative Non-Newtonian fluid was run. The chosen fluid was a well documented 41 g/L polymer solution of Dowanol DPM (dipropylene glycol methyl ether) thickened with a polymethyl methacrylate (PMMA) having a weight average molecular weight of  $2 \times 10^{\circ}$ .
- 29. As before, pressure drop versus shear rate plots were made at 40.0°C, this time using the three smallest diameter capillaries. The nominal diameters of these capillaries ranged from 0.30 mm to 0.50 mm with lengths of 25.40 mm. The plots were found to be linear with positive y-intercepts over the measured shear rate range from 2000 1/S to 80,000 1/S (Appendix D and Figure 7).

- 30. However, when the Bagley end correction was attempted, the results indicated both a positive or negative end correction length depending on shear rate, an anomalous result (Appendices E, F, G and Figure 8). This suggested the presence of slip and subsequently the slip correction was carried out. However, it too gave anomalous results in the form of a negative slip velocity for the lower shear rates (Appendices H, I and Figure 9).
- 31. The problem appeared to be both slip and end effect related and thus neither correction by itself was adequate. Thus, the only recourse was to alter the physical parameters,  $L_c$  and  $R_c$ , of the capillaries.
- 32. In an attempt to eliminate the slip, capillaries of larger diameter (D = 0.50 mm) were tried but due to their short length (25.40 mm) it became impossible to separate the very low pressure drops from other noise during a run. The manufacturer, Tinius Olsen Testing Machine Company, was then contacted but was unable to supply longer capillaries from stock. As a result, the experimentation was terminated until longer capillaries could be obtained or built. The new capillaries would have lengths of up to 50 mm and diameters greater than 0.50 mm. As the present furnace was designed primarily for capillaries of 25.40 mm in length, lengths of greater than 50 mm would present either problems in temperature control without major modifications to the present set up or a dramatic decrease in piston travel and hence test fluid capacity.

#### **CONCLUSIONS**

- 33. It was concluded that the Sieglaff-McKelvey Capillary Rheometer can accurately measure viscosities of Newtonian fluids as low as 1.5 poise and at shear rates from a nominal 1000 1/S to 100,000 1/S, and at temperatures as low as 5°C above ambient. The accuracy of these measurements was found to be better than 5% in every case.
- 34. It was also concluded that while it is possible to adapt the Sieglaff-McKelvey Capillary Rheometer to measure low viscosity polymer solutions at near ambient temperatures, such a conversion would require capillaries of greater length than those currently stocked by the manufacturer.
- 35. It was found that an increase in the signal to noise ratio of the piston load channel would allow the use of larger diameter capillaries by allowing the accurate measurement of very low pressure drops. Such an improvement would be especially

significant for Non-Newtonian fluids where large diameter capillaries are necessary to prevent slip from occurring. As a bonus, such an improvement would also expand the lower limits of the instrument's viscosity and shear rate ranges.

#### RECOMMENDATIONS

- 36. A number of recommendations are suggested to make the Sieglaff-McKelvey Capillary Rheometer more suited to the requirements of the DRES Physical Chemistry laboratory.
- 37. These include a modification to accept variable length capillaries such as that shown in Figure 10. A modified piston tip employing low friction Teflon o-rings to decrease both piston friction and leakage thereby improving the sensitivity of the piston load channel (Figure 11). A recirculating water/ethylene glycol environmental chamber might be designed to allow sub-ambient temperatures to be obtained, an impossibility with the present set-up. Finally, an electronic servo system to allow preprogrammed computer controlled runs could be built. A dedicated computer could also be used to handle data analysis directly off the instrument avoiding the present system of strip chart recordings and human interpretation, a lengthy and potentially error prone process.
- 38. With some or all of these modifications, the Sieglaff-McKelvey Capillary Rheometer would fulfill the initial requirements of the DRES Physical Chemistry laboratory.

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# APPENDIX A

# **Piston Dimensions**

RADIUS (R <sub>p</sub> ) (mm)	AREA (A <sub>p</sub> ) (mm <sup>2</sup> )
3.18	31.7
3.97	49.5
6.35	126.7
	3.18 3.97

# APPENDIX B

# Capillary Dimensions

DESIGNATION	RADIUS (R <sub>c</sub> ) (mm)	LENGTH (L <sub>c</sub> ) (mm)
Α	0.15469	25.40
В	0.20633	25.40
C	0.25432	25.40
D	0.31002	25.40
E	0.38788	25.40
F	0.49018	25.40
G	0.62131	25.40
Н	0.82201	25.40
J	0.82755	25.40
K	0.88960	25.40

APPENDIX C

Viscosity of Standardized Newtonian Oils at 40.0°C

η (NBS) (poise)	R <sub>c</sub> (mm)	L <sub>c</sub> (mm)	η (measured) (poise)	% error
200.3	0.15469	25.40	206.5	3.11
200.3	0.20633	25.40	199.8	- 0.27
67.44	0.15469	25.40	67.88	0.65
67.44	0.20633	25.40	68.20	1.13
67.44	0.25432	25.40	69.65	3.28
67.44	0.31002	25.40	68.57	1.68
13.89	0.15469	25.40	13.63	- 1.91
13.89	0.20633	25.40	13.70	- 1.73
13.89	0.25432	25.40	14.50	4.15
13.89	0.31002	25.40	14.03	1.03
4.203	0.15469	25.40	4.078	- 2.96
4.203	0.20633	25.40	4.063	- 3.32
4.203	0.25432	25.40	4.308	2.51
4.203	0.31002	25.40	4.320	2.89
1.494	0.15469	25.40	1.425	- 4.49
1.494	0.20633	25.40	1.439	- 3.69
1.494	0.25432	25.40	1.568	4.97

#### APPENDIX D

Shear Rate and Pressure Drop Data at 40.0°C Sample: 40.89 g/L Dowanol DPM/PMMA

Capillary A:  $R_c = 0.15469 \text{ mm}$   $L_c = 25.40 \text{ mm}$ 

SHEAR RATE (Γ) (1/S)	PRESSURE DROP (ΔP) (dyne/cm²)	SHEAR RATE (F) (1/S)	PRESSURE DROP (ΔP) (dyne/cm²)
1946	$1.381 \times 10^{6}$	20 068	$5.723 \times 10^6$
3000	$1.599 \times 10^{6}$	29 899	$7.993 \times 10^6$
3943	$1.887 \times 10^{6}$	41 047	$1.058 \times 10^{7}$
5078	$2.171 \times 10^{6}$	59 797	$1.492 \times 10^{7}$
10014	$3.442 \times 10^{6}$	79 257	$1.875 \times 10^{7}$
15000	$4.697 \times 10^{6}$		

Capillary B:  $R_c = 0.20633 \text{ mm}$   $L_c = 25.40 \text{ mm}$ 

SHEAR RATE (Г) (1/S)	PRESSURE DROP (ΔP) (dyne/cm²)	SHEAR RATE (F) (1/S)	PRESSURE DROF (ΔP) (dyne/cm²)
1943	$9.868 \times 10^{5}$	10 122	$2.518 \times 10^{6}$
2998	$1.172 \times 10^{6}$	15 290	$3.552 \times 10^6$
3989	$1.362 \times 10^{6}$	19 998	$4.737 \times 10^{6}$
5108	$1.626 \times 10^{6}$	29 982	$6.473 \times 10^{\circ}$
6065	$1.753 \times 10^{6}$	39 720	$8.427 \times 10^6$
7859	$2.289 \times 10^{6}$	60 648	$1.303 \times 10^{7}$

Capillary C:  $R_c = 0.25432 \text{ mm}$   $L_c = 25.40 \text{ mm}$ 

SHEAR RATE (Γ) (1/S)	PRESSURE DROP (ΔP) (dyne/cm²)	SHEAR RATE (F) (1/S)	PRESSURE DROP (ΔP) (dyne/cm²)
1966	$6.828 \times 10^{5}$	9 648	$2.052 \times 10^{6}$
2965	$8.762 \times 10^{5}$	15 050	$2.999 \times 10^{6}$
4060	$1.176 \times 10^{6}$	20 300	$3.963 \times 10^{6}$
4995	$1.263 \times 10^{6}$	24 997	$5.032 \times 10^{6}$
5976	$1.366 \times 10^{6}$	29 290	$5.624 \times 10^{6}$
8028	$1.760 \times 10^6$		

APPENDIX E

# **Bagley End Correction**

Sample: 40.89 g/L Dowanol DPM/PMMA (40.0°C)

Capillary A:  $R_c = 0.15469 \text{ mm}$   $L_c = 25.40 \text{ mm}$ 

SHEAR RATE (Γ) (1/S)	- L <sub>e</sub> /R <sub>c</sub>	L <sub>e</sub> (mm)	$\frac{L_e}{L_c} \times 100\%$
2000	49.6	- 7.67	- 30.2
3000	39.0	- 6.03	- 23.8
4000	29.4	- 4.55	- 17.9
5000	20.6	- 3.18	- 12.5
6000	12.6	- 1.95	- 7.67
8000	- 1.60	0.24	0.97
10 000	- 13.6	2.10	8.28
20 000	- 54.8	8.48	33.4
30 000	- 78.6	12.2	47.9
40 000	- 94.1	14.6	57.3
50 000	- 105.0	16.2	63.9
60 000	- 113.1	17.5	68.9
70 000	- 119.4	18.5	72.7
80 000	- 124.3	19.2	75.7

APPENDIX F

# **Bagley End Correction**

Sample: 40.89 g/L Dowanol DPM/PMMA (40.0°C)

Capillary B:  $R_c = 0.20633 \text{ mm}$   $L_c = 25.40 \text{ mm}$ 

SHEAR RATE (Γ) (1/S)	$-L_e/R_c$	L <sub>e</sub> (mm)	$\frac{L_c}{L_c} \times 100\%$
2000	49.6	- 10.2	- 40.3
3000	39.0	- 8.05	- 31.7
4000	29.4	- 6.07	- 23.9
5000	20.6	- 4.25	- 16.7
6000	12.6	- 2.60	- 10.2
8000	- 1.60	- 0.33	1.3
10 000	- 13.6	2.81	11.1
20 000	- 54.8	11.3	44.5
30 000	- 78.6	16.2	63.9
40 000	- 94.1	19.4	76.4
50 000	- 105.0	21.7	85.3
60 000	- 113.1	23.3	91.9
70 000	- 119.4	24.6	96.9
80 000	- 124.3	25.7	100.9

# APPENDIX G

# **Bagley End Correction**

Sample: 40.89 g/L Dowanol DPM/K125 EA (40.0°C)

Capillary C:  $R_c = 0.25432 \text{ mm}$   $L_c = 25.40 \text{ mm}$ 

SHEAR RATE (Г) (1/S)	$-L_e/R_c$	L <sub>e</sub> (mm)	$\frac{L_c}{L_c} \times 100\%$
2000	49.6	- 12.6	- 49.7
3000	39.0	- 9.92	- 39.1
4000	29.4	- 7.48	- 29.4
5000	20.6	- 5.24	- 20.6
6000	12.6	- 3.20	- 12.6
8000	- 1.60	0.41	1.6
10 000	- 13.6	3.46	13.6
20 000	- 54.8	13.9	54.9
30 000	- 78.6	20.0	78.7
40 000	- 94.1	23.9	94.2
50 000	- 105.0	26.7	105.1
60 000	- 113.1	28.8	113.2
70 000	- 119.4	30.4	119.6
80 000	- 124.3	31.6	124.5

#### APPENDIX H

# Slip Co-Efficient $(\beta)$

Sample: 40.89 g/L Dowanol DPM/PMMA (40.0°C)

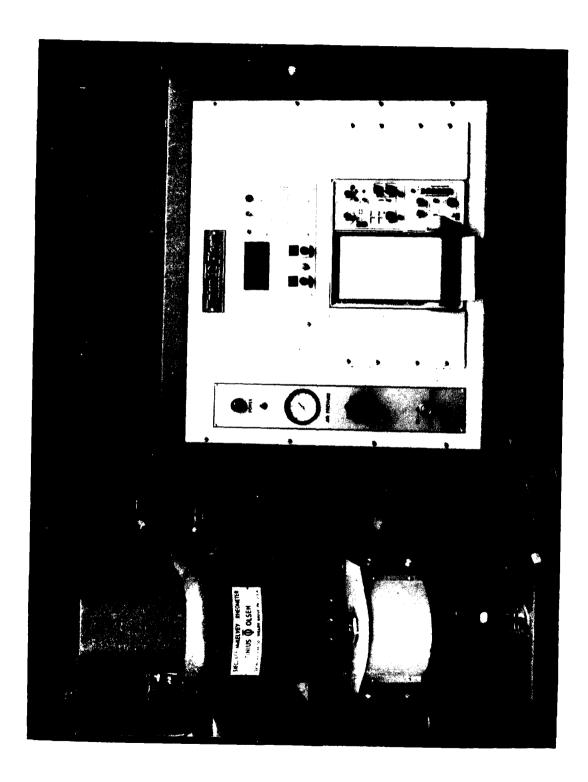
SHEAR STRESS $(\tau_{\omega})$ (dynes/cm <sup>2</sup> )	$\beta \left( \frac{\mathrm{cm}^3}{\mathrm{dyne} \cdot \mathrm{s}} \right)$	SHEAR STRESS $(\tau_{\omega})$ (dynes/cm <sup>2</sup> )	$\beta  \left( \frac{\text{cm}^3}{\text{dyne} \cdot \text{s}} \right)$
4 000	- 0.00403	30 000	0.00246
5 000	- 0.00253	40 000	0.00271
8 000	- 0.00029	50 000	0.00286
10 000	0.00046	60 000	0.00296
15 000	0.00146	70 000	0.00303
20 000	0.00196	80 000	0.00309

### APPENDIX I

# Slip/No Slip Shear Rates

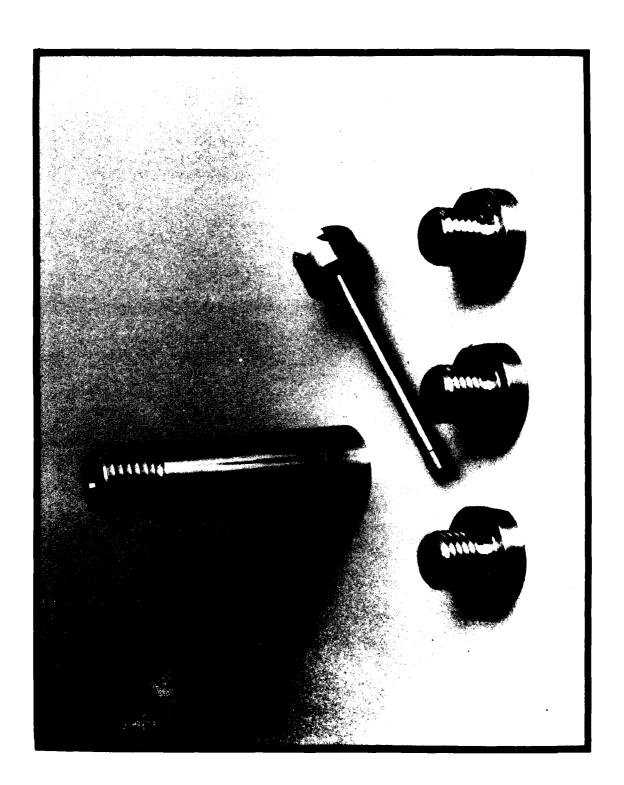
Sample: 40.89 g/L Dowanol DPM/PMMA (40.0°C)

NO SLIP SHEAR RATE (Γ) (1/S)	$(\Gamma_s)$ CORRESPONDING UNCORRECTED SHEAR RATE (1/S)			
	CAPILLARY A	CAPILLARY B	CAPILLARY C	
3000	84.4	347	672	
4000	1700	1900	2170	
5000	3320	3450	3660	
6000	4940	5010	5160	
8000	8180	7890	8150	
10000	11420	11220	11140	
20000	27600	26740	26110	
30000	43800	42270	41070	
40000	76180	73320	71000	
60000	92370	88860	85960	
70000	108600	104400	100900	
80000	124800	119900	115900	



SIEGLAFF-McKELVEY HIGH SHEAR RATE RHEOMETER SHOWING LOADING FRAME (LEFT) AND CONTROL AND RECORDING CONSOLE (RIGHT)

Figure 1



CYLINDER BARREL, PISTON AND CAPILLARIES FOR SIEGLAFF-McKELVEY RHEOMETER Figure 2

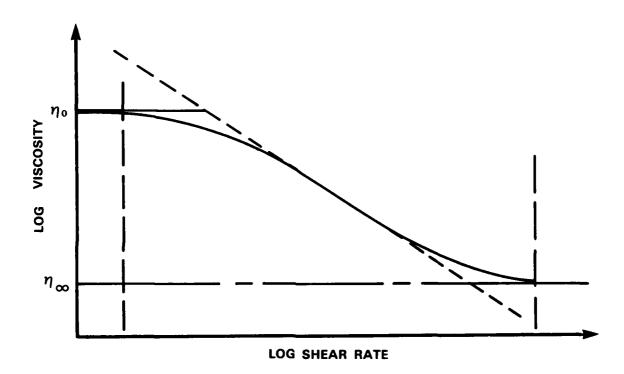
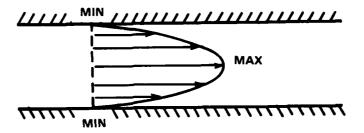
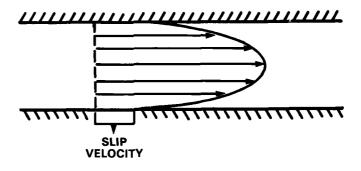


Figure 3 A PLOT OF LOG VISCOSITY VS LOG SHEAR RATE FOR A TYPICAL SHEAR THINNING POLYMER ILLUSTRATING ZERO SHEAR ( $\eta_o$ ) AND INFINITE SHEAR ( $\eta_\infty$ ) VISCOSITIES



VELOCITY PROFILE WITHOUT SLIP



**VELOCITY PROFILE WITH SLIP** 

Figure 4

CAPILLARY VELOCITY PROFILES WITH AND WITHOUT SLIP

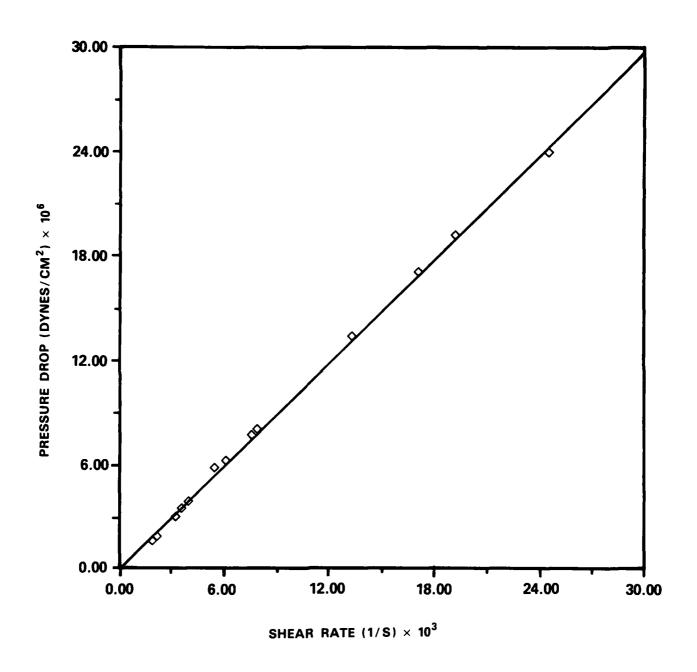


Figure 5

A TYPICAL PRESSURE DROP VS SHEAR RATE PLOT FOR A NEWTONIAN OIL. (CAPILLARY B; CANNON INSTRUMENT VISCOSITY STANDARD S 600, 4.203 POISE AT 40.0°C)

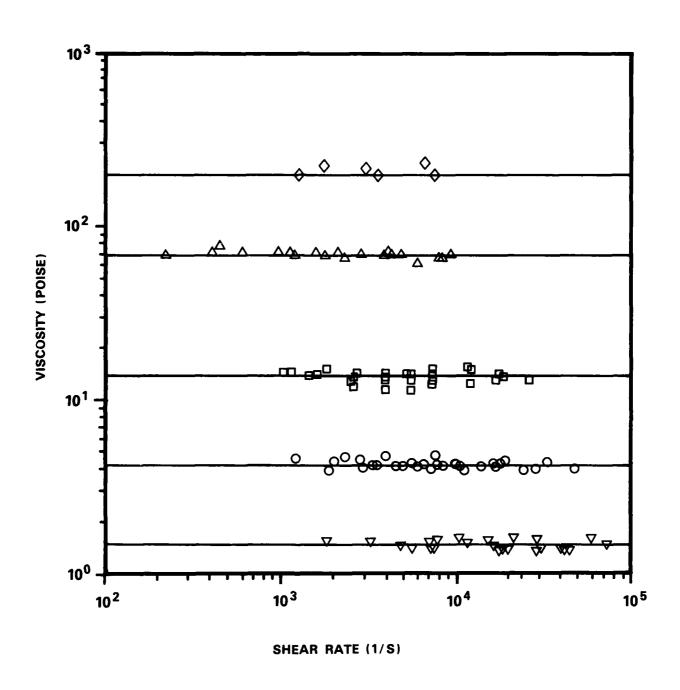


Figure 6

VISCOSITY VS SHEAR RATE PLOTS FOR FIVE NEWTONIAN OILS.

LINES REPRESENT NBS VISCOSITY WHILE POINTS REPRESENT ACTUAL MEASURED VISCOSITIES USING SIEGLAFF-McKELVEY RHEOMETER (SEE APPENDIX C FOR AVERAGE VALUES)

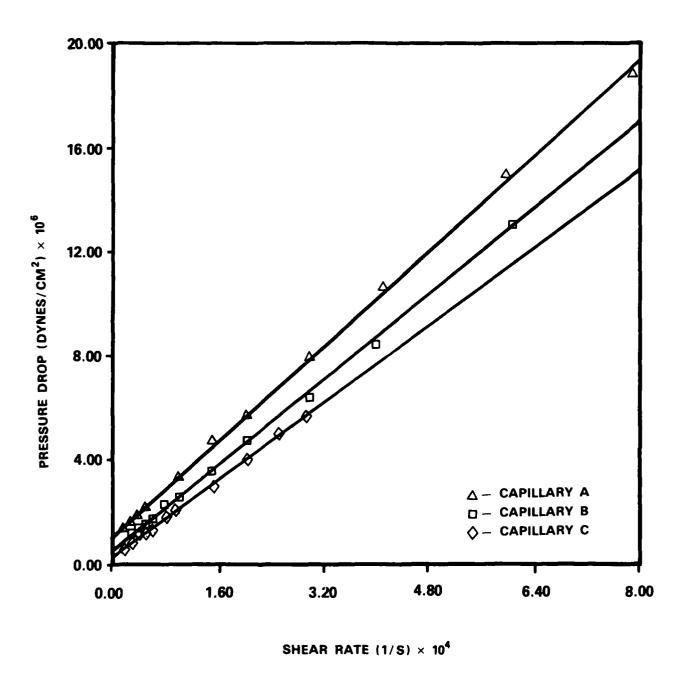


Figure 7

PRESSURE DROP VS NEWTONIAN SHEAR RATE PLOTS FOR

40.89 g/L DOWANOL DPM/PMMA AT 40.0°C

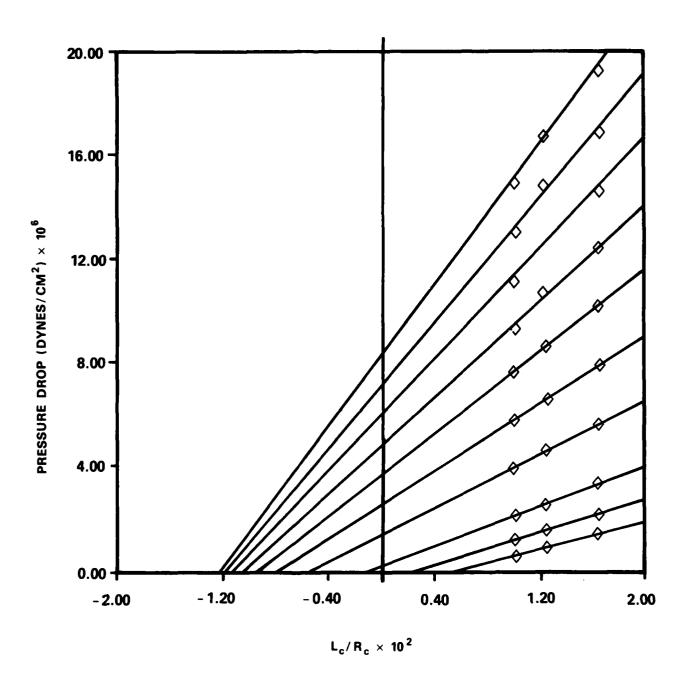


Figure 8 BAGLEY PLOT: PRESSURE DROP VS  $L_c/R_c$  FOR 40.89 g/L DOWANOL DPM/PMMA (40.0°C) AT TEN DIFFERENT SHEAR RATES. BY CALCULATING THE X-INTERCEPT THE VALUE OF  $L_e/R_c$  CAN BE DETERMINED.

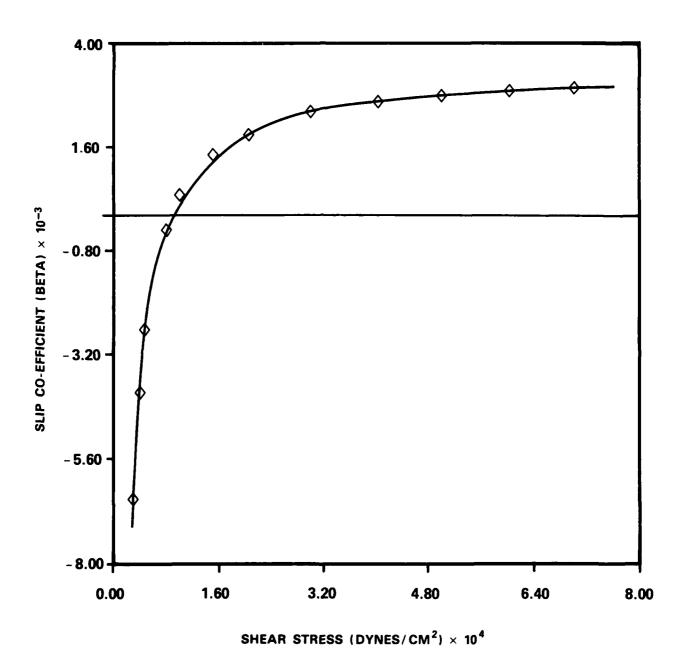
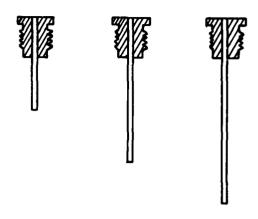


Figure 9

SLIP CO-EFFICIENT (BETA) AS A FUNCTION OF SHEAR

STRESS FOR 40.89 g/L DOWANOL DPM/PMMA AT 40.0°C



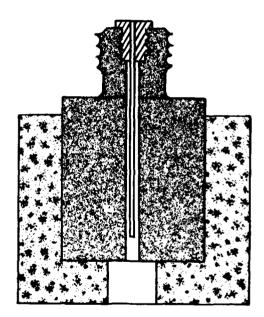




Figure 10

CURRENT CAPILLARY DESIGN (LEFT) AND VARIABLE
LENGTH CAPILLARIES AND INSULATED HOLDER (RIGHT)

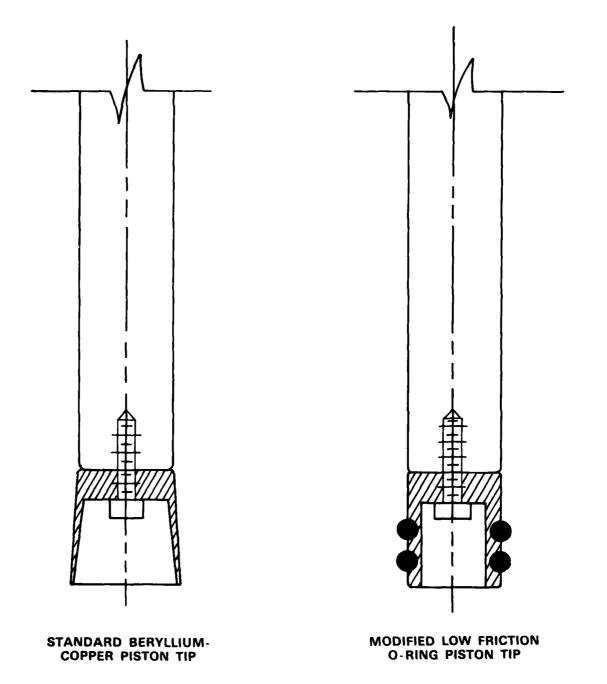


Figure 11

PISTON TIPS: STANDARD (LEFT) AND PROPOSED O-RING TYPE (RIGHT)

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#### 13. ABSTRACT

An investigation was made into the adaptation of a Sieglaff-McKelvey high shear rate melt rheometer for use in measuring low viscosity fluids. Using calibrated Newtonian oils, viscosities as low as 1.5 poise were found to be measurable with better than 5% accuracy over a shear rate range from a nominal  $1000\ 1/S$  to  $100,000\ 1/S$  at temperatures as low as  $5^{\circ}C$  above ambient. However, difficulties were experienced in the measurement of low viscosity polymer solutions. With the stock capillaries 25.40 mm in length, a combination of wall slip and entrance/exit effects made calculating true viscosities impossible. It was concluded that capillaries with a diameter of 1.0 mm or greater and variable lengths of up to 50 mm would be required to eliminate slip and still obtain a pressure drop within the measurable range of the instrument. To facilitate accurate measurement of low pressure drops, a new piston design was proposed using low-friction, low leakage teflon o-rings. This would also expand the lower limits of the instrument's viscosity and shear rate ranges. (U)

#### KEY WORDS

high shear rate viscometer Sieglaff-McKelvey viscometer Newtonian liquids polymer solutions rheology

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